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January 6, 2002

Mr. Bruce Morrison
U.S. Environmental Protection Agency (EPA)
901 North 5th Street
Kansas City, Kansas 66101

Subject: **Lead Speciation Study–December 9, 2002 Meeting Minutes**
EPA Region 7, START 2 Contract No. 68-S7-01-41, Task Order 0027
Herculaneum Lead Smelter Site
Task Monitor: Bruce Morrison, EPA Project Manager

Dear Mr. Morrison–

On December 9, 2002, a conference call was held to discuss comments issued on the Lead Speciation Study performed by Drs. David Johnson and Jerrold Abraham of the State University of New York under Task Order 0027. Enclosed you will find the December 9, 2002, meeting minutes, which provid an overview of the topics discussed and decisions made. You may contact me at 913-495-3922 with any questions regarding these minutes.

Sincerely,

Angela N. Suárez
START 2 Analytical Services Coordinator

for Hieu Q. Vu, PE, CHMM
START 2 Program Manager

cc: Gene Gunn, EPA
Jim Silver, EPA
Ryan Schuler, Seagull Envirotech, Inc.
Shelley Rice, Tetra Tech EM Inc.

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MEETING MINUTES
HERCULANEUM LEAD SMELTER PROJECT
LEAD SPECIATION STUDY
DECEMBER 9, 2002

Participants:

Bruce Morrison, Project Manager, U.S. Environmental Protection Agency (EPA) Region 7
Gene Gunn, Federal Facilities/Special Emphasis Branch Chief, EPA Region 7
Jim Silver, On-Scene Coordinator, EPA Region 7

David Johnson, PhD, Professor of Chemistry, State University of New York (SUNY)
Jerrold Abraham, M.D., Director of Environmental and Occupational Pathology, SUNY

Ryan Schuler, Superfund Technical Assessment and Response Team (START) Project Manager, Seagull Environmental Technologies, Inc. (Seagull)

Angela Suárez, START 2 Analytical Services Coordinator, Tetra Tech EM Inc. (TtEMI)
Hieu Q. Vu, PE, CHMM, START 2 Program Manager, TtEMI

Meeting Information: The meeting was a conference call in which participants joined by calling 866-270-2016 and entered meeting ID 6515. The call began at 9:00 AM central standard time (CST) and was approximately 1 hour and 20 minutes in duration.

Meeting Minutes:

- The meeting began with a short introduction of the participants followed by general meeting instructions. Mr. Bruce Morrison, EPA, informed the participants that they would be discussing the comments on the lead speciation study and the format of the report.
- The meeting followed the general outline of the November 20, 2002, comments from the Doe Run Company (Doe Run) and October 22, 2002, comments from the State of Missouri Department of Natural Resources (MDNR). Each comment from these two documents was discussed in turn. The actual comments are presented below in blue italics to supplement the meeting minutes.
- The first comments to be discussed were those issued by Doe Run.
- Mr. Morrison, EPA, stated that Doe Run was the most challenging entity to deal with in regards to answering all comments fully. He further stated that two other experts had reviewed the speciation study (Dr. Scott Clark of University of Cincinnati and Dr. David Sterling of St. Louis University) and endorsed the method fully.

Doe Run Comment 1: *The Study makes an incorrect assumption that lead paint sources are only represented by Ti-Pb particles (p. 16). Although recognizing that lead-based paints are "mainly present" from several compounds, including "lead carbonate" (p.16), there is no further discussion of this particular compound. Due to the age of housing in Herculaneum within ½ mile of the smelter and in "concrete town" (Thurwell and Hill streets) being mainly pre-WWII (with a*

significant portion built prior to 1930), a significant lead paint source will be lead carbonate from the early high-lead "white lead". Uses of titanium in lead paint occurred later and had lower concentrations of lead. The result of this false assumption is that a significant amount of lead from lead carbonate and other lead paint sources has probably been inappropriately included in the authors' general "Other" category. Clearly, the Study should have been designed to evaluate lead carbonate.

- Dr. David Johnson, SUNY, began discussing the comments. In regards to Doe Run Comment 1, he stated that lead carbonate cannot be distinguished from lead oxide. Lead with zinc and lead with titanium are indicators of lead-based paint. It is possible that lead-based paint particles are found in the soil, but it is not likely; however, the possible presence of lead-based paint particles cannot be excluded.
- Mr. Morrison, EPA, pointed out that Doe Run had been using fertilizers with phosphorous for many years on some of the residential soils. He said that this may contribute to the lead phosphate formation in the soil.
- Mr. Gene Gunn, EPA, asked what could have been done differently with the study to account for the presence of lead-based paint.
- Mr. Johnson, SUNY, replied that it would have been very difficult to change anything, but there is a reference that he would add to the list of references supporting the report. Additionally, he stated that an expert could have looked for the lead-based paint particles, the large particles could have been sectioned off and analyzed using an electron microprobe, and a differential IPA technique could have been used where the individual particles are treated with micro-droplets of acid
- Mr. Gunn, EPA, stated that no samples were collected from the smelter itself. He asked that if the smelter had been sampled, would this have helped the study.
- Mr. Johnson, SUNY, stated that this would have helped by expanding the scope of work, but you still would not be able to distinguish between lead oxide and lead carbonate without the methods mentioned previously.
- It was determined that collecting a sample from the smelter may be possible.
- Mr. Hieu Vu, TtEMI, asked if background sampling would be helpful in determining the presence of lead-based paint. He suggested that a sample be collected from non-residential soils, which theocratically should not contain lead-based paint particles.
- Dr. Johnson stated even if the background sample was collected that this would still not exclude the presence lead-based paint.
- Mr. Gunn, EPA, asked if they could learn anything from the smelter sample.
- Dr. Johnson, SUNY, replied that they could.
- Dr. Abraham, SUNY, asked that if the smelter sample would be from current emissions. He was informed that the smelter sample would be from current emissions. He further stated that it would be uncertain as to how well the current emissions would relate to what was present in the soils because of the time difference, but reiterated that this would be more information that could be used.

Doe Run Comment 2: *The Study erroneously classifies all lead > 60%Xri as "Oxide". (p.15 &16) This is most evident from the Study's Table V. Sample H17, which should contain 100% concentrate, is listed as 72.9% concentrate and 26.3% "Oxide". That a quarter of the concentrate was inappropriately labeled "Oxide," is also reflected in Figure 6a. Roughly one quarter of what the authors acknowledge is "Galena" has lead .60%XRI and, therefore, is classified by the authors as "Oxide", not "Concentrate". The misclassification of concentrate as "Oxide" may lead to inappropriate conclusions of houses with "Oxide" where the lead in the samples might be the very low bioavailable galena.*

- The second comment to be discussed, Doe Run Comment 2, referred to sample H-17. Doe Run stated that the sample (which is a concentrate sample) should be 100% concentrate. Dr. Johnson clarified that this is assuming that this is a legitimate sample of concentrate. He further stated that they were looking for unique particles in the concentrate that were not present in the other sources. He stated 73% of the estimated particle mass is from particles unique to the smelter. The other particles are from non-distinguished sources. He continued by saying that they can look at the ratio of distinct particles to "oxides". If this ratio is consistent, then this can be used in any later mathematical treatment of the data. He concluded by stating that the ratio of 3:1 that was developed may not be constant, because only one sample was collected and analyzed.

Doe Run Comment 3: *The Study erroneously classifies slag. (p. 16) Although Sample H16 and its replicate RH16 should contain 100% slag, the four values in the Study's Table IV &V (two samples each at <13 um and <100 um) are only 22.0%, 21.1%, 38.6%, and 77.0%, with corresponding "Oxide" values of 38.0%, 20.8%, 14.5%, and 4.2%, and corresponding "Other" values as 40.0%, 58.1%, 47.1%, and 18.9%. Thus it is clear that a significant portion of the "Oxide" categories may actually reflect very low bioavailable slag.*

- In response to Doe Run Comment 3, Dr. Johnson stated that the same applies for the slag as it does for the concentrate; neither contain 100% unique factors. Dr. Johnson reiterated that they were working on the ratio of unique to non-unique particles.
- Mr. Morrison, EPA, addressed a question to Mr. Ryan Schuler, Seagull, about the sample locations for the slag and concentrate.
- Mr. Schuler, Seagull, replied that the concentrate was collected from the primary dump area after the haul truck had been emptied and that the slag sample was collected from the upper layer of the slag pile.
- Dr. Abraham, SUNY, asked if the samples looked homogeneous when they were collected.
- Mr. Schuler, Seagull, replied that they did look homogeneous.
- Dr. Abraham, SUNY, asked if any study had been done on the composition of the slag pile. He was informed that no study has been done.
- Mr. Morrison, EPA, stated that the smelting process has changed over the years.

Doe Run Comment 4: *The Study erroneously assumes that all the "Other" category is "derived from some mixture of the general road dust category and the local external soil." (p. 19) As already pointed out above, the "Other" category probably contains lead from concentrate, slag, and paint sources, as well as from actual road dust and soil. Without any explained justification, the Study states that "House Dust" equals "Road Dust" minus "Paint" minus "Soil". The discussion above refutes this assumption. The position that all non-identifiable lead comes from the road is totally unsupportable in light of other studies. Refer for instance to the Sterling handling of unidentified source material.*

- In response to Doe Run Comment 4, Dr. Johnson, SUNY, stated that for the source apportionment that they worked with what they had. They tried to apportion the lead to the sources that were given to them. If more sources had been given, then the apportionment could have been more complete. The "other" category could have been apportioned from a variety of different sources.
- Mr. Gunn, EPA, stated that the report did indicate uncertainty in the closed end solution. He further stated that possibly one source that was left out of the original study was the emissions from the smelter stack.
- Dr. Johnson, SUNY, stated that they were given no information about the variability of the sources and were unable to determine the variability in the sources by the samples they were given.
- Mr. Morrison, EPA, asked how many samples from each source were recommended in order to determine the variability.
- Mr. Johnson, SUNY, replied that he believed that 3-4 samples from each source site would be beneficial. He also pointed out an additional problem of the time line for deposition. He didn't know if the current sources were necessarily related to the samples that were collected. He recommended doing a horizontal and vertical study of the slag pile to determine its composition.
- Mr. Gunn, EPA, stated that the purpose of the study is for attribution and enforcement. He continued by stating that the study needs to show that the particles found inside the homes are attributable to Doe Run. He stated that it would be nice to eliminate lead-based paint as a possible source, but that this was not a key issue.
- Dr. Johnson, SUNY, stated that he believed that it would be more cost effective to have an electron microscopy expert who is experienced with particle recognition and experts who can deal with multiple particle recognition. He stated that they are unable to address this comment by Doe Run. He stated that he has to admit the uncertainties and that this is circumstantial evidence at best. He stated that he is not a professional expert witness.
- Mr. Morrison, EPA, stated that he doesn't know of any historical data concerning the lead particles at the site, but he did know that lead has been emanating from the smelter, that there was a possibility that lead has been changed in the soil and that the soil may have been tracked into the homes. He asked if they can definitively say what has happened in the past.
- Dr. Johnson, SUNY, stated that this could not be done with the present data, but if they could find a stable particle that hasn't changed, then this may be able to be done. He further stated that

not having historical data makes it difficult to attribute this to current data. The attic samples may be helpful, but the attic samples are not necessarily concrete.

- Mr. Morrison, EPA, stated that he is being greatly challenged at this time to get some type of concrete evidence.
- Dr. Johnson, SUNY, stated that there are ways to improve the study. The use of experts who can identify lead-based paint particles is one of these ways. He stated that he knew of two experts: (1) Dr. Andrew Hunt, who started this study but left halfway through to form his own electron microscope company, and (2) Russ Krutcher of Microprobe Laboratories Northwest. Dr. Johnson also stated that if he had known the background involving the study, he would have liked to have seen where the samples were collected. It would have been good to have the experts involved in the sampling effort.
- Mr. Morrison, EPA, asked if Dr. Drexler is an expert in these areas.
- Dr. Johnson, SUNY, replied that he believes Dr. Drexler to be an expert in the microprobe and chemical areas, but he doesn't know if Dr. Drexler knows anything about environmental risks or apportionment.

Doe Run Comment 5: *The Study inappropriately concludes that "Road Dust" is the main source of Herculaneum House Dust. (p. 20) As discussed above, the equations used to determine Road Dust are based on false assumptions. That this conclusion is false is also illustrated by reviewing the one sample of "Road Dust" actually taken. Apart from issues of the number and location of such samples (see comment below), it should be evident from Table III that the lead concentratoin, particle diameter, and particle geometric mean for sample H18 (the only sample of "Road Dust" used in the Study) reflects significant physical differences from the various House Dust samples given (samples H3, H6, H8, H9, H11, H12, and H13). For example, the Study concludes that Road Dust constitutes 100% of the House Dust sample H9. However, the amount of total lead in House Dust sample H9 is almost twice the amount in the Road Dust sample H18.*

- In response to Doe Run Comment 5, Dr. Johnson, SUNY, stated that it is possible to extrapolate road dust with stable signature particle types, if these are the only sources. He does think it is reasonable to conclude that house dust is mostly from road dust.
- Dr. Johnson, SUNY, explained his theory on how lead was deposited in the homes. He explained that he doesn't believe there are any additional sources, but is handicapped in the fact that he wasn't able to actually visit the site.
- Mr. Gunn, EPA, stated that all that can be done now is to collect current road dust samples.
- Mr. Morrison, EPA, stated that there is monthly air monitoring data for the past 10 years that may be worth looking at.
- Doe Run Comment 6 was blacked out of copy that was sent to Dr. Johnson of SUNY.

Doe Run Comment 7: *The road samples collected recently do not represent the road samples of most of the history of the smelter. They can be used for prospective projections but not for historical analysis.*

- Doe Run Comment 7 was already addressed.

Doe Run Comment 8: *We need a more complete understanding of the methods used. However, it appears that no source samples were taken from the plant emissions. Because that source was not considered it is not possible for it to be identified as a source in the study. In the SIP mass balance approach, this methodology was employed successfully.*

- Dr. Johnson, SUNY, stated that Doe Run Comment 8 could be remedied by writing the methods clearer.
- Dr. Johnson, SUNY, stated that he did not understand Doe Run Comment 9. Mr. Morrison, EPA, told Dr. Johnson not to worry about this comment.

Doe Run Comment 10: *Attic contamination is not a pathway in most cases and therefore not relevant to this process.*

- Dr. Johnson, SUNY, replied to Doe Run Comment 10 by saying that the attic contamination is another source of contamination, not a pathway for household dust.
- Doe Run Comment 11 was discussed briefly.
- Doe Run Comment 12 was disregarded.
- Dr. Johnson, SUNY, didn't see any issues with regards to Doe Run Comment 13.

Doe Run Comment 14: *Even though slag is ubiquitous in Herculaneum, according to the Walker and Associates report, it is apparently not getting into the households according to the SUNY report. This is consistent with the findings in Port Piere, Australia, some years ago. The risk models should take this into consideration in the transfer of slag from the yard into the house.*

- In response to Doe Run Comment 14, Dr. Johnson, SUNY, stated that there was no indication of slag in the household dusts. He agrees with this comment.

Doe Run Comment 15: *The SUNY report assumes that all of the non-identifiable lead comes from the road. There is no scientific justification provided for this position; it is apparently just an assumption. Refer for instance to the Sterling handling of unidentified source material in his paper is St. Francios County.*

- Dr. Johnson, SUNY, stated in regards to Doe Run Comment 15 that this was a consequence of concentrating the solution in the apportionment attempt to only those sources available. He had no other remarks on this comment.
- Mr. Morrison, EPA, instructed Dr. Johnson, SUNY, to disregard Doe Run Comment 16.
- The conversation then turned to discuss the comments issued by the MDNR.

MDNR Comment 1: *Appendix I was not included in my copy. This appendix is necessary to understand the methods used.*

- Dr. Johnson, SUNY, stated in response to MDNR Comment 1 that Appendix I should have been provided to MDNR. Mr. Morrison, EPA, stated that this should have been done, but wasn't.

MDNR Comment 2: *In Section 2, Analytical Methods, the acronyms CCSEM and IPA need to be defined.*

- Dr. Johnson, SUNY, stated that they could address MDNR Comment 2.

MDNR Comment 3: *In Section 3, Results, why aren't Pb oxides presented?*

- Dr. Johnson, SUNY, stated that he didn't know the answer to MDNR Comment 3 and that he did not understand the question. Mr. Gunn speculated that the author of these comments associated lead oxide with paint instead of using the more appropriate lead carbonate.

MDNR Comment 4: *Table II–The column labeled "Bulk Pb (% PA) In Samples" doesn't make any sense. This should be "Sample ID".*

- Dr. Johnson, SUNY, stated that MDNR Comment 4 was due to a typo, and that this comment would be addressed.

MDNR Comment 5: *Figure 2d. Why is Al vs. Si plotted? A graph of Pb vs. CO_3 would be much more useful information.*

- In response to MDNR Comment 5, Dr. Johnson stated that there Figure 2d was not a plot of Al vs. Si. Figure 2c, however shows this plot. Al vs. Si was plotted to show if there was any Al or Si present. Dr. Johnson stated that Figure 2c shows there is a presence. Dr. Johnson further stated that they can't do a graph of lead verses carbonate.
- Dr. Johnson, SUNY, that all these comments could have been better explained if Appendix I was included with the report.

MDNR Comment 6: *Figure 6b. I don't understand why S vs. Pb is presented in the oxide and carbonate fractions? If they could segregate this it should have been presented as stated above.*

- In response to Comment 6, Dr. Johnson, SUNY, stated that the computer controlled scanning electron microscopy (CCSEM) technique is a semi-quantitative technique. He stated that this particular plot shows information about the software used and that there is a lead/sulfur X-ray overlap correction factor that can't be tinkered with. He stated that the program determines the overlap correction and that there are limitations to the CCSEM technique in this respect. They opt for less conservative lead/sulfur correction to maximize the identification of the real lead/sulfur particles because of the high presence of galena.
- Dr. Johnson, SUNY, went on to explain figures 3a, 3b, 6a, and 6b.

MDNR Comment 7: *Section 3.1.4, Lead Particle Types in Herculaneum, again the analysis is incomplete without assessing Pb carbonate and oxide particles.*

- Dr. Johnson, SUNY, stated in response to MDNR Comment 7 that there is no way of distinguishing lead carbonate and lead oxide particles using the CCSEM technique. They can call them high lead compounds, but they can't determine which is which.

MDNR Comment 8: *Section 3.2.1. The assignment of Pb> 60 as oxides is arbitrary. Why isn't carbonate classified?*

- Dr. Johnson, SUNY, stated that the same applies to MDNR Comment 8. The assignment of lead may be arbitrary but it is based on standards.
- Mr. Morrison, EPA, stated it may be better to say circumstantial instead of arbitrary.
- Dr. Johnson, SUNY, stated that it could be circumstantial based on the circumstances of the methods used, but would prefer to refer to it as imperial.

MDNR Comment 9: *Table IV. The very high percentage of particles qualifying as "Other" indicates classification system isn't working. Carbonates and oxides need to be classified.*

- In response to MDNR Comment 9, Dr. Johnson, SUNY, explained that the classification scheme is based on the nature of the samples and the nature of the analytical instrument. He stated that there is a reason for the "other" category. It shows that there is a significant population of unique particles, but that the number of populations themselves are small. He disagrees with the comment that the classification scheme is not working. He believes that it is working. The "other" category could be broken into the particles they represent, but nothing would be contributed to the attribution scheme if this was done.

MDNR Comment 10: *Section 3.3. There is little justification to ascribe the "Other" category to road dust. The contribution from smelter fallout is totally overlooked.*

MDNR Comment 11: *Section 3.3.2, again, the authors need to identify smelter-derived particles.*

- Dr. Johnson, SUNY, stated in response to MDNR Comments 10 and 11 that there was no information presented to him about fallout. He stated that it would be helpful if he could get information about the settled dust.

MDNR Comment 12: *Section 3.3.3, page 20, it is difficult to support the conclusion with the data. At top of page 21, how do they distinguish alumino-silicate matrix from smelter source?*

- In response to Comment 12, Dr. Johnson, SUNY, stated that Figure 4c shows the tracer flag for the slag sample. This figure could be improved if there were more slag and dust samples collected and analyzed.
- Mr. Morrison, EPA, instructed Dr. Johnson, SUNY, that the other sets of comments were redundant or unimportant and that they did not need to be addressed at this time.
- Mr. Gunn, EPA, asked if Dr. Johnson, SUNY, had any response to a comment made about the use of a mass spectrometer (MS).
- Dr. Johnson replied that there are other alternatives to the CCSEM method for the speciation of lead. He stated that the MS could follow the use of a laser ablation technique to gather a tremendous about of information about the particles. However, there are very few laboratories that employ this technique, the technique is time consuming, and a limited fraction size of particles can be seen.
- Mr. Morrison, EPA, stated that this technique has been used on a number of his other sites and was unaware of the limitation of particle size.
- Dr. Johnson, SUNY, stated that you can see smaller particle sizes when you use a SEM technique and then transfer these particles to another type of analytical method.
- Dr. Johnson, SUNY, recommended that to improve the study they need to assemble optical microscope, ICPMS and SEM analysts on site to determine the challenges they would face.
- Mr. Gunn, EPA, asked if there were any responses to the sieving issues mentioned in the comments. He asked why such small particle sizes were used.
- Mr. Johnson, SUNY, stated that the smaller particle sizes were used for two reasons: (1) the smaller particles would more likely stick to the hands of children and (2) the smaller particle sizes tend to be more homogeneous, which provides for a better analytical technique.
- Mr. Gunn, EPA, asked if because such small particle sizes were being used, if any of the paint data were lost.
- Dr. Johnson, SUNY, replied that they would find larger chips in larger particle sizes, but that there would also be degraded paint particles found in the smaller particle sizes. He agreed that some paint data was lost.
- Mr. Gunn, EPA, asked if the CCSEM technique could be applied to larger particles.
- Dr. Johnson, SUNY, replied that it could, but that it would double the analytical effort.
- Mr. Gunn, EPA, asked if considering all limitations and maintaining that one can argue with the percentages, can it be concluded that at least some material from the smelter is getting into the homes.
- Dr. Johnson, SUNY, stated that he would be able to say Yes.

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- Mr. Gunn, EPA, asked if there could be any changes made to the study to improve it. He would like to see this information in writing.
- Dr. Johnson, SUNY, stated that there were several things that he could think of: (1) increase the number of source samples, (2) allow for a site visit to gain a better understanding of the situation, (3) allow more time and funding to analyze samples in greater detail, (4) do micro-manipulation of particles, such as differential IPA, and (5) assemble experts of different fields to discuss the situation.
- Mr. Gunn, EPA, asked how big of an effort it would be to assemble the group of experts.
- Dr. Johnson, SUNY, replied that it would be approximately 2-3 days of someone's time plus anywhere from 3,000 to 6,000 miles of travel, or more. It would initially cost between $6,000 and $10,000 to establish the first talks.
- Mr. Gunn, EPA, asked how much it would cost for the execution phase.
- Dr. Johnson, SUNY, estimated that it would be 5 to 10 times what was spent initially.
- There were no further comments to discuss and so the meeting ended with tasks being assigned.
- Mr. Vu, TtEMI, asked Mr. Gunn, EPA, if there was a need to put together an introduction package concerning the past, current, and future activities of the site.
- Mr. Gunn, EPA, said that this was a good idea and it needed to be done.
- Mr. Gunn, EPA, also stated that TtEMI would be responsible for arranging any follow-up with the professors for the actions to be taken.
- The meeting concluded at approximately 10:20 AM CST.